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SPACE PROCESSING FLOAT ZONE THERMAL ANALYSIS

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The Boeing Company Seattle, Washington

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1.0 INTRODUCTION

Growth of single crystals in vertical arc-imaging furnaces has been successfully demonstrated by various researchers. Early experiments with these furnaces using a carbon-arc energy source, encountered difficulties in maintaining uniform light intensity for required periods of time. Development of high-power Xenon arc lamps substantially reduced the instability problem experienced with carbon-arcs, and also allowed longer-term operation for crystal Experiments have shown that one of the preferred approaches for growing single crystals in arc-imaging furnaces is the pedestal technique. This technique has alternatively been labeled a modified Czochralski, or modified float-zone process. principal advantage of the float-zone process over other crystal growth techniques is production of higher purity crystals; achieved by eliminating the need for a crucible and the resulting crucible contamination. The homogeneity of crystals grown by the float-zone process, as with other processes, is affected by circulation patterns in the molten semi-conductor material. Carruthers and Grasso have extensively studied the stability and fluid flow in floating zoner in both a simulated zero-gravity environment and on Skylab. Effects of convection patterns on homogeneity of solute distribution and electrical resistivity have been reviewed by Benson. considerations should apply to float-zone crystal growth, and indicate that for the Czochralski process, all convection in the melt should be avoided.

Growth of single crystals in space is expected to substantially improve the solute homogeneity of crystals, by reducing or eliminating convection within the melt region. It should also be possible to form larger diameter crystals because the length of the suspended liquid zone can be increased in the absence of gravity.

Maintaining a large-diameter uniform-temperature melt zone will be a key problem in designing a crystal growth experiment using a solar concentrator. Accordingly, this study has been directed at thermal modeling of the liquid zone to predict temperature gradients for various parametric changes.

2.0 ANALYSIS

Two generalized lumped parameter nodal math models were formulated to represent a right circular cylindrical, BETA-CYL, and a body of revolution configuration, BETA-BOR, with time varying boundary conditions. The physical system was sectioned into an array of discrete volumes and the mass of each volume element was "lumped" at a point within the volume which it represented. The paths for heat transfer from one mode to another were represented by conductors joining the appropriate nodes. The thermal models each contained a maximum of 300 nodes, Figure (1) depicts the right circular cylindrical nodal math model.

The thermal math models described above were input into the Boeing Engineering Thermal Analyzer Program (BETA). This program solves an electrical analog network system using an iterative numerical method. Both transient and steady state solutions are treated. A logic diagram of the computer program detailing the computational steps is given in Figure (2). Several BETA subroutines required development to permit numerical simulation of the applied heat flux boundary conditions and the change of phase process.

The heat pulse subroutine is illustrated in Figure (3). This permits the specification of an externally applied heat flux of the form:

$$q_i = q_0 e^{-A|X-Vt-B|^2}$$

where:

q - Peak surface heat flux, Btu/min-in²

X - Distance from reference end of crystal, inches

V - Pulse velocity away from reference end of crystal, in/min

t - Time elapsed since start of pulse movement, min

B - Starting position of center of pulse measured from reference end of crystal, inches

A - Pulse width parameter

Values of A of 100 and 4 were used, the relative widths of the heat pulses can be seen in Figure 4.

A similar cooling subroutine was also formulated to represent surface cooling.

During a phase change each node must receive or release a quantity of energy equal to its latent heat of fusion. This energy exchange accounting system was simulated by a phase change subroutine, PHAS. Figure (5) illustrates the logic system. Each node is surveyed to see if it is at or undergoing a phase change. If it is undergoing a phase change then an accounting system keeps the nodal temperature constant at the melting temperature until the energy exchange matches the node latent heat of fusion.

This study did not directly consider the transparency of silicon to thermal radiation; however, the "no convection" and "Infinite convection" extremes considered for energy transport in the moltan zone could be expected to bound the effects which might be observed had internal radiation been considered. Furthermore, with a cylincrical crystal, any radiation interchange between the surroundings and crystal internal regions due to transparency can only tend to reduce radial temperature gradients and consequently give a more uniform axial flow of thermal energy which is required to obtain flat interfaces. Radiation interchange in the axial direction, between internal crystal regions, was not expected to be very significant as temperature differences within the crystal are much less than temperature differences which exist between the crystal and its surroundings.

3.0 DISCUSSION OF RESULTS

A total of 82 runs were made to de-bug, checkout, and perform production analyses for this study. Only the runs shown in Table 1 contained significant results. The other runs, although more numerous, were less significant in terms of expended machine time.

The first block on this table describes the crystal configuration for each run and refers to a right circular cylinder unless otherwise stated. The right circular cylinder crystals were considered to consist of 30 wafers of equal thickness as defined by planes normal to the cylinder axis (see "Wafer Thickness" column).

The surface heat flux refers to the peak surface heat flux of a Gaussian shaped pulse when the word "Gauss" appears in the "Pulse Width" column. If the word "Uniform" appears in the "Pulse Width" column, the surface heat flux value applies over the entire heated length, and the number appearing in the "Pulse Width" column is the heated length, in inches, of a section of cylinder centered along the cylinder axis.

For some runs, the peak surface heat flux, \mathbf{q}_0 , was reduced linearly with time; the \mathbf{q}_0 limits where applicable, are described in the "Surface Heat Flux" column. In any case, when the run time, i.e. "Real Time" in the "Time Data" block, exceeds the time shown in the "Heat Time" column, the heat is reduced to zero after the "Heat Time" shown.

In the "Number of Nodes" column, "300" indicates that each wafer was considered to consist of 10 rings, the temperatures of which were computed as node temperatures by the program. The number "120" indicates each wafer was considered to consist of 4 rings.

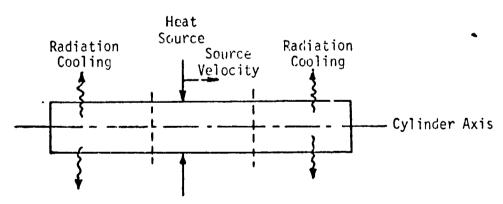
Under the "Cooling Parameters" block, the "Sink Temperature" and "Script F" define radiation rates from the radial surface of the crystal for the axial length of surface specified in the "Cooled Area" column. The ends; normal to the cylinder axis, were considered insulated for all runs.

Except for Runs 35, 72, 75, 80, 81, and 82, the initial temperature, uniform throughout the crystal, was one degree Fahrenheit below the melting point. This was done to prevent wasted computer time raising the crystal to the melting point. Runs 72, 75, 80, 81, and 82 were intended to determine steady state conditions and estimates of the final temperatures were used as initial temperatures for the analysis to conserve machine time, i.e., initial temperatures above the melting point were used in the heated region, and temperatures somewhat below the melting point were used in the cooled region.

Run 35 was a check of the program against an analytical solution for a cylinder, with uniform constant surface heat flux, using an initial uniform temperature of zero; excellent agreement was obtained.

3.1 Cylindrical Melt Zones

The initial part of this study consisted of analyzing cylindrical specimens (BETA-CYL program) on a transient basis using a constant strength heat source moving in the axial direction with ends cooled by radiation to a 70°F environment as shown below:



The source of the incident radiant energy was not modeled since it was considered that it could be best treated after determining the boundary conditions required to produce flat interfaces. The following parameters were varied in the transient analyses: heated length, cooled length, specimen diameter, constant source strength (moving heat sources), and transient source strength (moving and fixed heat sources). It was determined that an adiabatic section

between the heated and cooled sections would be beneficial in producing flat interfaces. Additionally, it was found that excessive machine time was required with a transient-type analysis due to the rather low solidification rate required for growth of high-perfection crystals (\approx 1 cm/hr). A preferable analytical technique, in comparison to the transient analysis, for treating the continuous zone refinement/growth along a rod was to obtain the steady-stage solutions for configurations with stationary boundary conditions which give flat interfaces. If an extremely slow feed rate of the specimen is then introduced after steady state is achieved, a negligible change in the shape and location of the interfaces would occur with respect to a fixed reference system. Machine time required to solve for steady-state conditions is nominal provided that reasonable estimates of the final conditions are provided as inputs.

Figure 6 describes the conditions and results for Run 71. Excellent flat-interface solidification conditions were obtained and are shown in Figure 7. The physical state of each ring of each wafer has been represented in the computer printout by an "0", "1", or "2". Each column of figures represents a wafer of the crystal as defined in Table 1. The radial width of each wafer, as defined in the program, varies so that the volumes of each ring are equal regardless of the nominal ring radii. The figures are, therefore, not to scale in the radial direction. Since all wafers are the same thickness in the axial direction (for the right circular cylinder configuration) the printouts are to scale in the axial direction. The axis of the cylinder is indicated by a centerline at each end of the crystal. A "O" indicates that the state of that node is solid. A "l" indicates a molten state, and "2" indicates a partially melted state at the melting temperature. A column of zeros adjacent to a column of twos or ones indicates a flat solidification interface within a one-waferthickness tolerance. These printouts were used as a visual tool to allow rapid scanning of run results for each case. More exact definition of interface shapes can be determined by inspection of node freezing fraction data in the computer result numerical output.

The data printed at the left of each state are:

Time - minutes from start of analysis

 $G_{A_{MAX}}$ - maximum axial temperature gradient, °F/inch

G_{RMAX} - maximum radial temperature gradient, °F/inch

T_{MAX} - maximum temperature, °F

A melting point temperature of 2573°F was assumed for silicon. Conductivity and specific heat values employed as a function of temperatures are shown in supplementary Tables 2 and 3.

Run 71 was for a 1-inch diameter crystal, 7.5 inches long with a stationary uniformly irradiating heat source applied to the central 2 inches of specimen. The source strength decayed linearly from 20 Btu/min-in² to 0 in the first 2 minutes of run time. For approximately 7 minutes after the heat source was shut off, the central molten zone solidified with virtually flat interfaces. The solidification rate was approximately 40 cm/hr, which is higher than current state of the art. The solidification process, however, could be increased to any desired duration by reducing the cooling rate at the crystal ends, e.g., through the use of louvers or radiation shields. The simulated high solidification rate was used here to conserve machine time. A lower cooling rate would be accompanied by lower temperature gradients in the crystal compared to those shown on Figures 6 and 7.

respectively. A section through one half of the length of crystal is shown with each node defined by a rectangle (to scale), with resulting steady state temperatures given at each node in degrees F. The melt interface shape was sketched in based on freezing fraction data from the numerical output of the program.

A nearly converged solution using the steady state analysis technique was obtained with the case analyzed in Run 75; the corresponding conditions and results are shown on Figure 8. Although there was a slight deviation from flatness at the interface, it is believed that this could be eliminated by a slight reduction in the rate of



source irradiation which would move the interface into the insulated (shielded) region where the thermal flux lines are more nearly straight.

To bound the effects of convection in the molten zone, an "infinite convection" analysis was performed for the same configuration as in Run 75. This condition was implemented by assigning a cup-mixing temperature to the melt at each iteration which has the effect of instantaneously distributing throughout the melt zone all incident energy at the surface. The results are shown in Run 82, Figure 9. A virtually flat interface was obtained in almost the same position as with no convection. By comparing figures 8 and 9 it is seen that there is little difference in the two solutions except that the melt temperature with convection is uniform at a value somewhat lower than the peak value observed with no convection. The degree of convergency to a steady state solution can be assessed by noting how closely the total cooling rate approaches the constant heating rate. For Runs 75 and 82 the errors are 3% and 1% respectively, which is sufficient convergence for preliminary purposes.

Additional transient results, Runs 29, 36, 39, 40, 44, 49, 55, 56, 60, 62 and 65, similar to those presented for Run 71 are shown in Figures 10 through 20. The significant results for each of these runs are shown in the "Comments" column of Table 1.

Crystal specimen dimensions and boundary conditions are shown on Figures 4, 21, and 22 for Runs 49, 55, 56, 60, and 62. Of particular interest are the heat pulse shapes and positions on the specimen as a function of time which cannot be readily visualized from Table 1 data.

For Runs 60, 62, and 65 maximum axial and radial temperature gradients, maximum temperature, and peak surface heat flux are shown as a function of time on Figures 23, 24, and 25 respectively. The irregularities in the temperature and gradient curves are due to (1) the results of a lumped parameter thermal analysis, e.g. at the instant when a node melts or solidifies, a relatively sudden adjustment in adjacent node temperatures occurs, and (2) the location of the peak temperature and gradients moves around

in the crystal, whereas, the plots show only the peak values regardless of location in the crystal.

3.2 Body of Revolution Melt Zones

The body-of-revolution program (BETA-BOR) has the capability to analyze configurations where the specimen has a shape more complex than a right circular cylinder. Possible configurations of interest are shown on Figure 26: (a) vertical cylinder float zone with gravity (b) float-zone in Zero-G with rotation, and (c) crystal growth from different sized feed stock. An example of case (c) was analyzed for 6-inch and 12-inch rods meeting in a molten zone with an assumed curved section transition; the configuration and boundary conditions are shown on Figure 27. Estimates of steady state conditions used for program input are shown in Figure 28. Conditions after 2.35 minutes are shown on Figure 29. Steady state convergence is approached with a good flat solidification interface on the 12-inch end. The interface shapes were sketched in on Figure 29, based on melt fraction data from the program output for the respective interface nodes.

Figure 27 shows the locations of the nodes for Run 80 as limited by the ability of the node size to be resolved into cells on the printout page. The dashes and asterisks are used in an alternate fashion for each wafer to define the node boundaries for each wafer. Each wafer has 10 ring shaped nodes, however on the 6" diameter end, not all are shown due to the resolution limitation described above. The radial interfaces between the nodes in regions of body diameter change are considered by the program to be conical surfaces. The uniform irradiation rate shown for the heated area is based on actual surface area, not a correct area based on some assumed ray direction. The shape shown on the printout is approximately to scale.

Figure 28 shows the initial conditions for Run 20: an asterisk indicates a temperature above the melting point, a dash indicates a temperature below the melting point. The difference between this figure as compared to Figure 7 is that the symbols at each node correspond to the node state rather than alternating to define node boundaries. Final conditions are shown for Run 80 on Figure 29

after the indicated elapsed time, given the Figure 28 initial conditions. Dashes and asterisks are as described for Figure 28 and an "X" symbol denotes a node partially melted at the melting temperature. Melt interface shapes shown are based on freezing fraction data for the melting nodes. Temperatures at selected points were shown for reference. Heating and cooling rates were shown as a guide to the deviation from steady state, i.e. if a true steady state had been reached, heating and cooling rates would be equal. This condition could be achieved by allowing the run to proceed in time, at a corresponding cost in machine time. Figures 30, 31, and 32 show data similar to that of Figures 27, 28, and 29 which apply to Run 72; neither steady state conditions nor flat interfaces were obtained.

CONCLUSIONS

Thermal math models of a silicon float zone crystal growth process were formulated. Transient temperatures and melt zones were determined for a variety of simulated external heat flux conditions. It was found that nearly flat interface solidification conditions could be simulated by varying the applied heat flux, as in cases 65 and 71. Additionally, convective affects were found to be negligible in reducing temperature gradients.

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wation	Wafer thickness	Ē	2.0	2.	5	63	63	20	0.2	70	03	6.2	6	\$2	0 25	0.25	0.75	8.0	63	ã	9.
Cylinder configuration	ŝ	ŝ		-	3	6	•	•	•	•	•	•	•	7.5	7.5	7.5	7.5	7.5	7.5	BOR 6" to 12"	BOR 6" to 12"
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condition is metting point minus one degree except runs 35, 75, 81, 82, 72, and 80

See Table 1 discussion in text for pulse width d

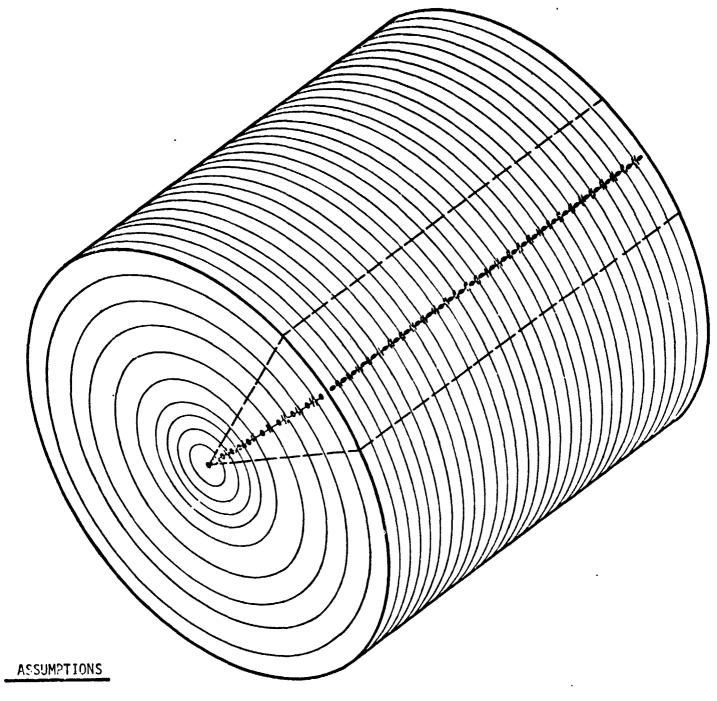
SILICON THERMAL COMDUCTIVITY TABLE ?

TEMP - 1	Thermal Conductivity	Btu/min-in-°F
32	.1348	
80.3	.1187	
170.3	.0955	
20.3	.0793	
440.3	.0611	
620.3	.0497	
800.3	.0408	
980.3	.0339	
1160	.0287	
1340	.0250	
1520	.0223	
1880	.0196	
2440	.0178	
2573	.0176	
10 ⁶	.0176	

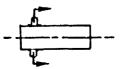
SILICON SPECIFIC HEAT

TABLE 3

TEMP - °F	Specific Heat - Btu/LB-°F
0	.157
200	.184
400	.197
600	.203
800	.208
1000	.211
1200	.216
. 1400	.220
1600	. 224
1800	.228
2000	. 234
2200	.242
2400	.245
2573	. 249
2600	. 250
106	.250

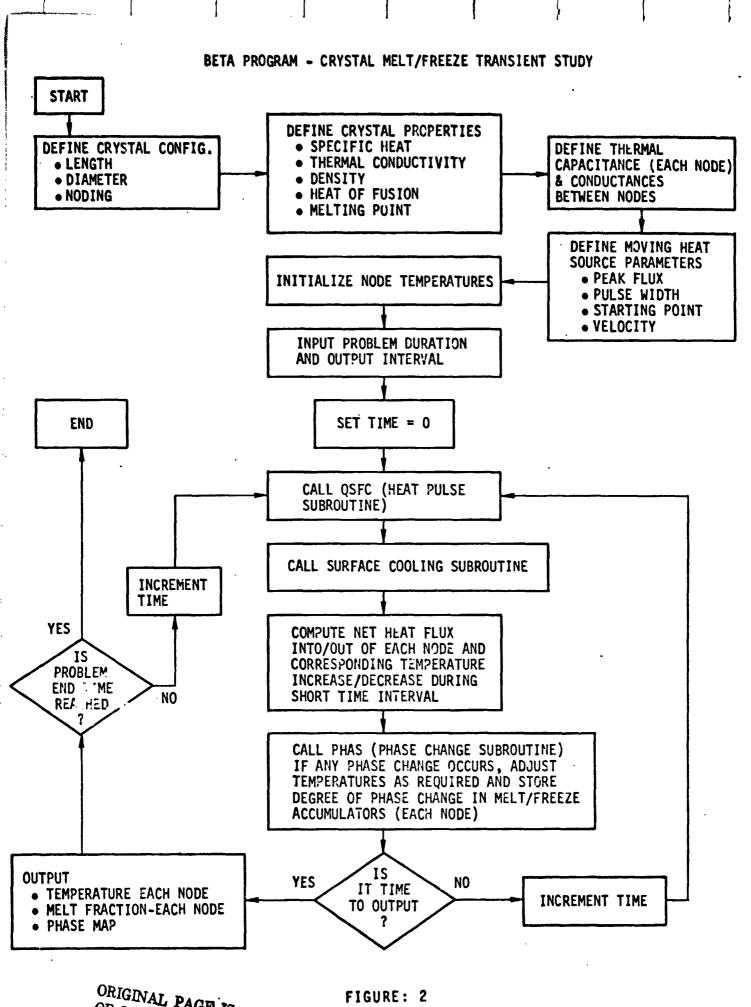


- CENTER CORE + 9 RINGS FOR EACH WAFER 30 WAFERS
- MASS OF EACH RING OF EACH WAFER IS LUMPED TO FORM NODE (300 NODES)
- ENDS ARE INSULATED
- MOVING GAUSSIAN SURFACE HEAT FLUX DISTRIBUTION



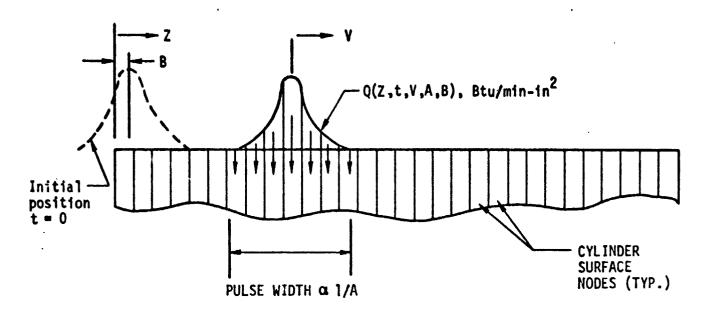
• SURFACE COOLING TO SURROUNDINGS BY RADIATION

FIGURE: 1 MATH MODEL



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HEAT PULSE SUBROUTINE (QSFC)



CALL QSFC (Q₀,t,V,A,B,Q₁) GO TO FIRST SURFACE NODE COMPUTE HEAT INPUT TO NODE i: Q_i = \pi DQ₀ \int \frac{Z_2}{Z_1} e^{-A(Z-vt-B)^2} dz NO THAT THE LAST SURFACE NODE? YES RETURN TO BETA PROGRAM COOLING SUBROUTINE IS SIMILIAR

FIGURE: 3

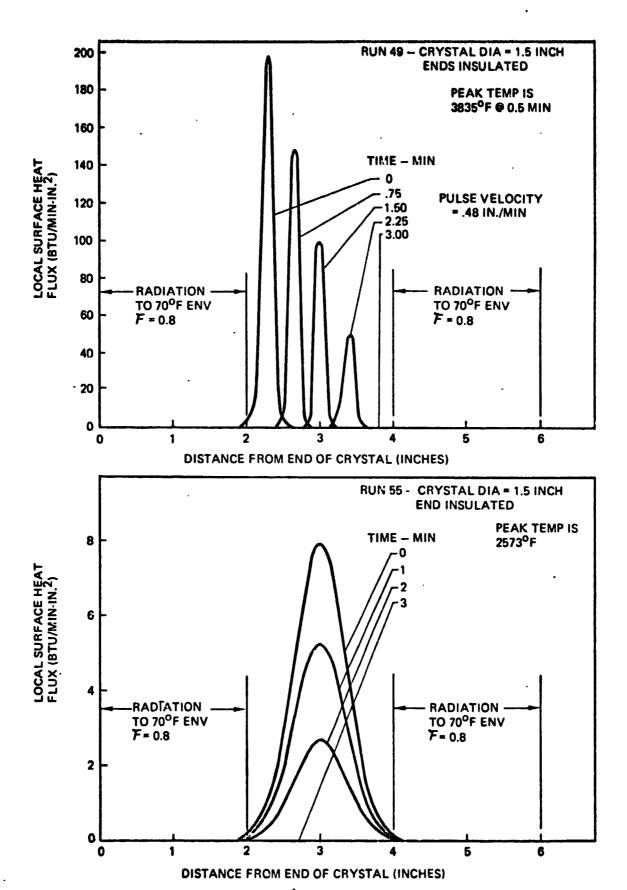


Figure 4

RUN 49 & 55 - LOCAL SURFACE HEAT FLUX PROFILES

PHASE CHANGE SUBROUTINE (PHAS)

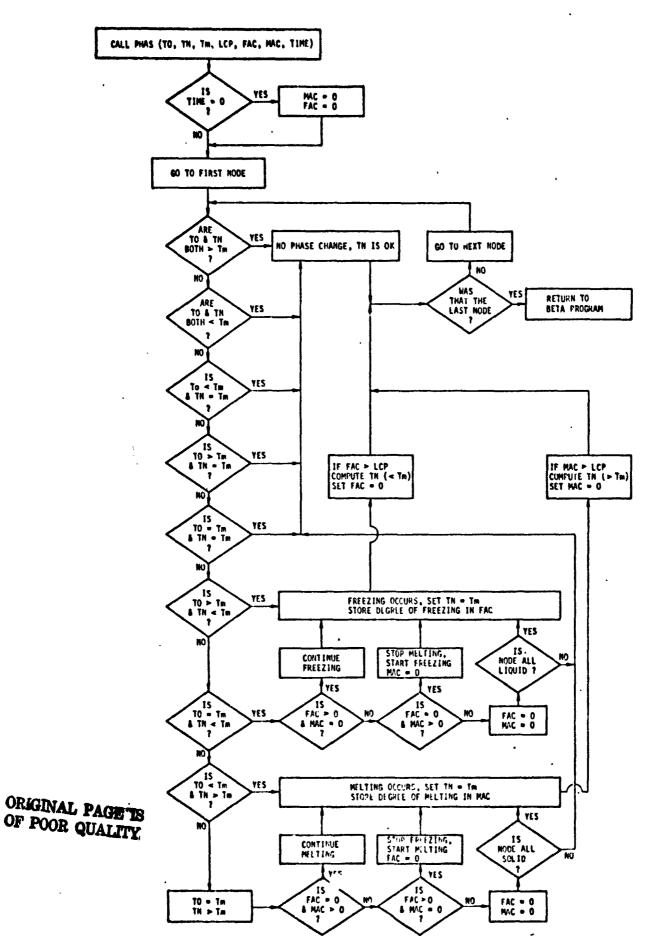
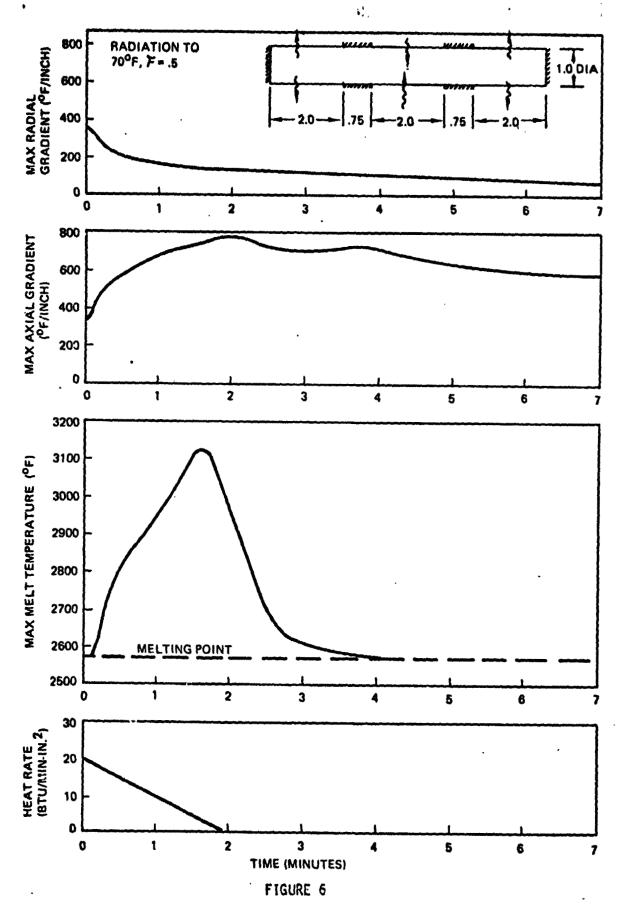


FIGURE: 5

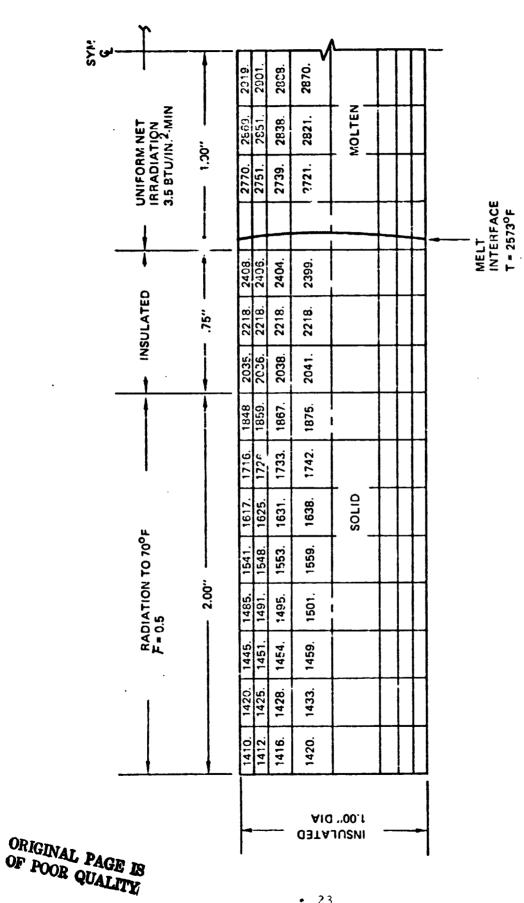


RUN 71 - TEMPERATURE GRADIENT & HEAT FLUX PROFILES

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Construction Cons	GA = 464	GA = 553 CC0C3CC0000111111100300013390 CC0C000C002032222200000 Tmax = 2749, CCC000C0020000000000000000000000000000	GA = 639

Figure 7. Run 71 - MELT ZONES

Figure 7 Run 71 (Continued)



STEADY STATE-NO CONVECTION Figure 8. Run 75

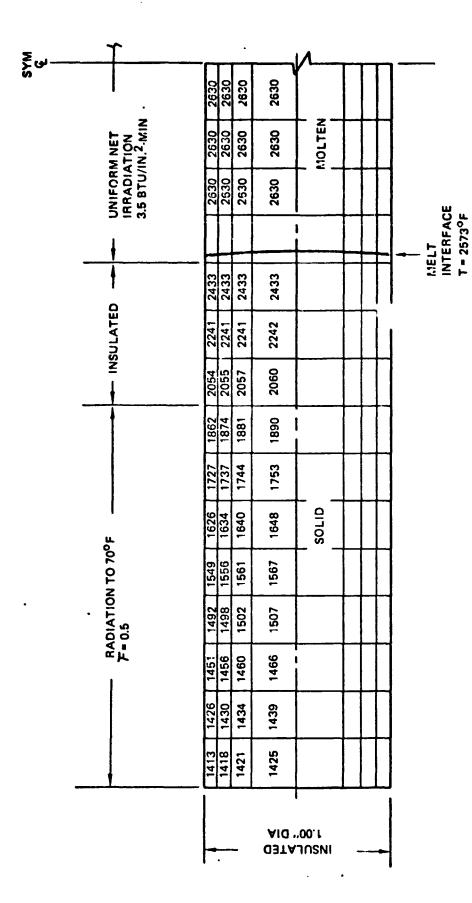


Figure 9. Run &2 STEADY STATE-INFINITE CONVECTION

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FIGURE: 10 . Run 29 - MELT ZONES

	
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Figure 10. Run 29 (Continued)

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MELT ZONES . Run 36 FIGURE: 11

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FIGURE 11. Run 36 (Continued)

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FIGURE: 12 Aun 39 - MELT ZONES

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FIGUFE: 13 . Run 40 (Continued)

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FIGURE: 14 . Run 44 - MELT ZONES

Figure 14. Run 44 (Continued)

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Figure 15. Run 49 - MELT ZONES

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Figure 15. Run 49 (Continued)

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Figure 17. Run 56. - MELT ZONES

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Figure 17. Run 53 (Continued)

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Figure 17. Run 56 (Continued)

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TIME 0.02500	00000000000000000000000000000000000000	TIME= 0-27500	CCCCOUNCOCOZZZIIZZZ 0000000000 00C000000000000000000000000	11MFs 0.60000	CCCOCCCOCCCTITITZOCOCCCCOCOCCCCCCCCCCCCC	TIME 0.92812	CCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCC	TIME- 1-30000	CC000000000111111000000000000000000000

Figure 18. Run 60 (Continued)

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Figure 19. Run 62. - MELT ZONES

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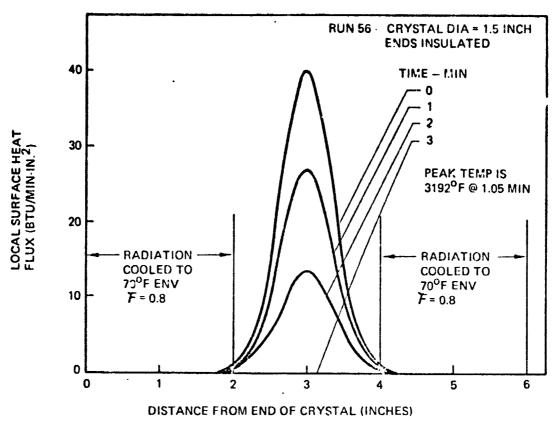
Figure 19. Run 62 (Continued)

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16490.0 agait .		TIME# 1.13750		TIME 1.59844		TIMEs 2.73594		TIPE 2.89844	
	60000000022222200000000000000000000000		000000000011111111123000000000000000000		6CGCGGGGGGGGGT11111111100GGGGGGGGGGGGGGGG		6C0C0000001111111100000000 CC0CC0000000211111220000000 CC0CCCC0000002211112200000000 CC0CCCC000000211111220000000000		CCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCCC
TIME. 0.02127		TIME. C.24062			i	TIME# 0.90312	i	71HE- 0-78750	

Figure 20. Run 65 - MELT ZONES

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11 # 11			TIME				
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TTHE- 2-91875		713E - 2.9390	** TIPE 2.95937		TIME 2.97969		

Figure 20. Run 65 (Continued)



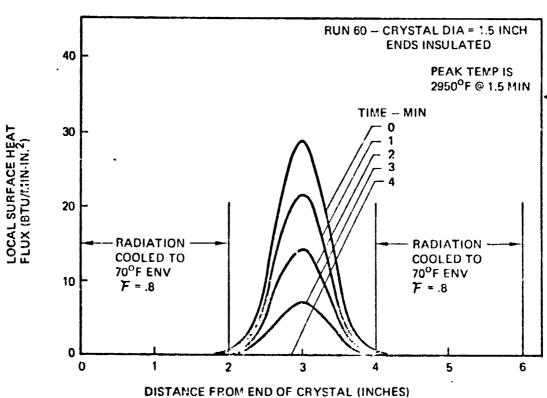


Figure 21
SURFACE HEAT FLLX RUN 56 & 60

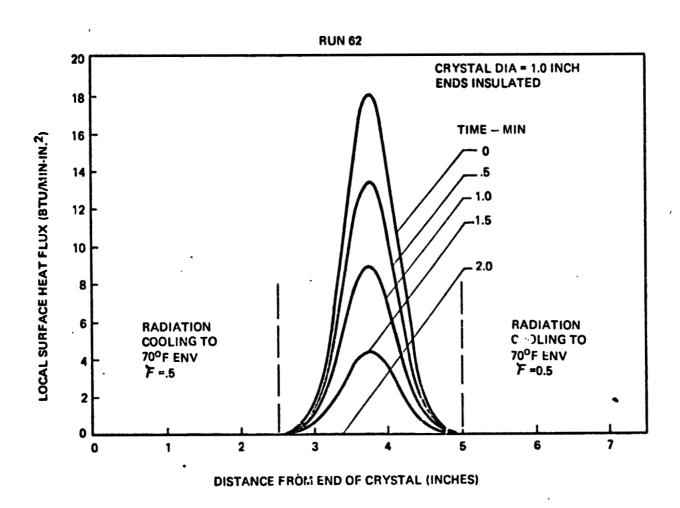


Figure 22
SURFACE HEAT FLUX RUN 62

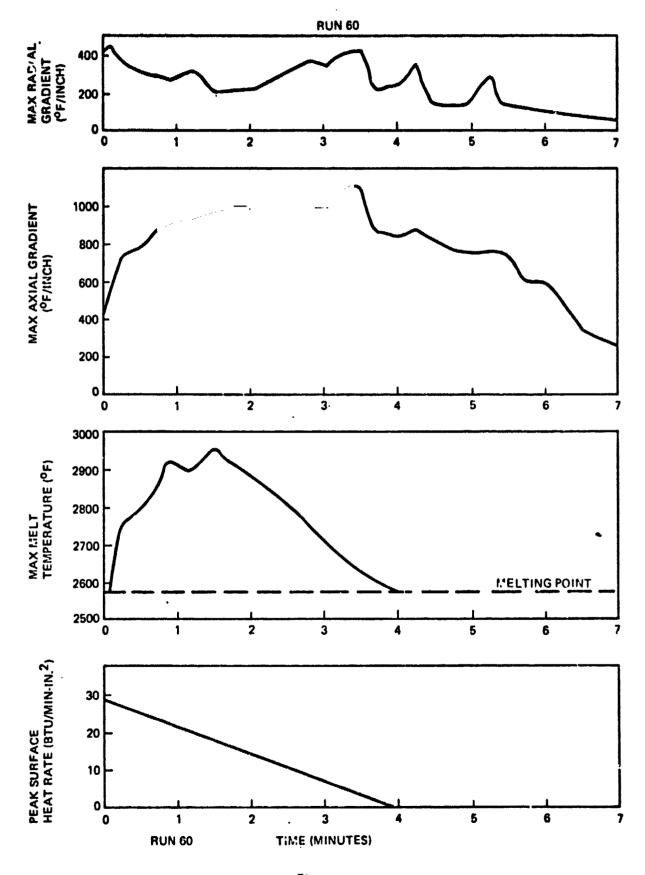


Figure 23
RUN 60 - TEMPERATURE GRADIENT & HEAT FLUX PROFILES

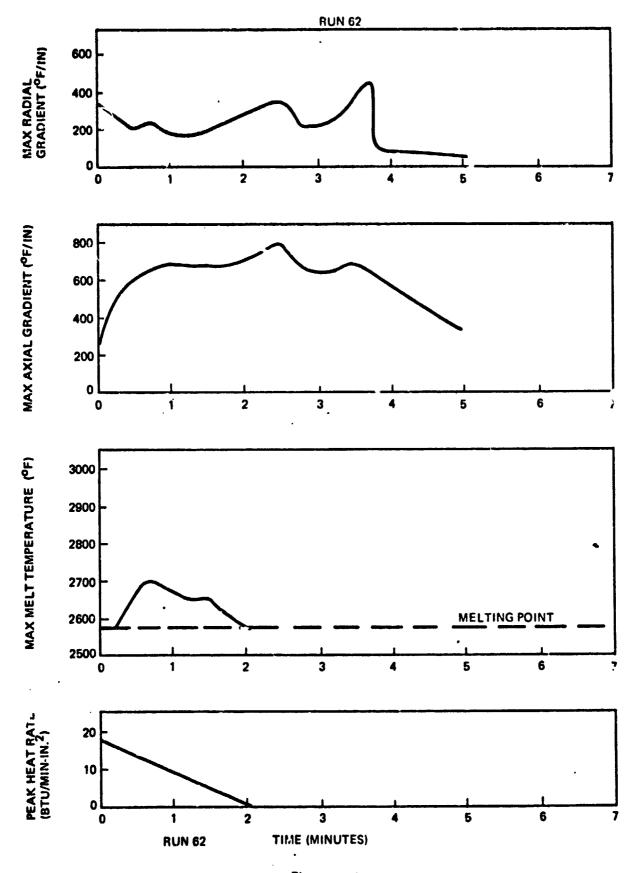
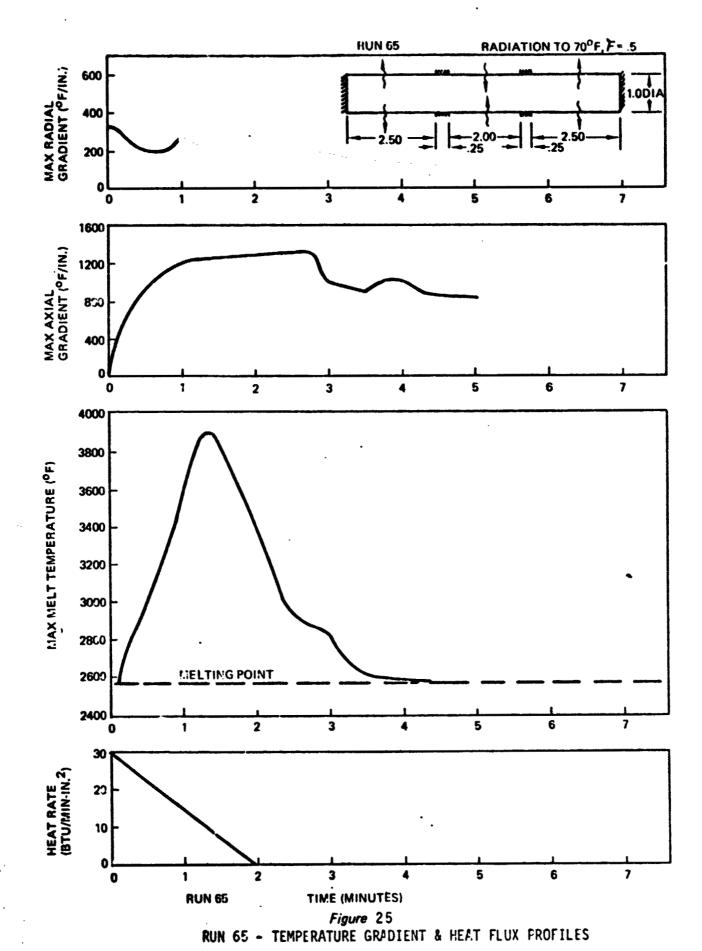
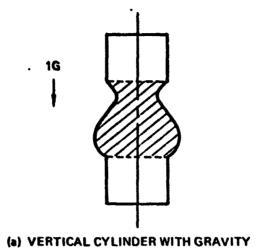
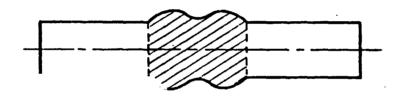


Figure 24

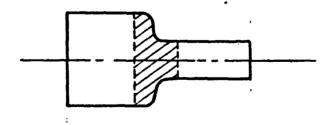
RUN 62 - TEMPERATURE GRADIENT & HEAT FLUX PROFILES
50







(b) FLOAT ZONE IN ZERO-G WITH ROTATION



(c) FLOAT ZONE BETWEEN DIFFERENT DIAMETERS

FIGURE: 26. Typical Body-of-Revolution Configurations

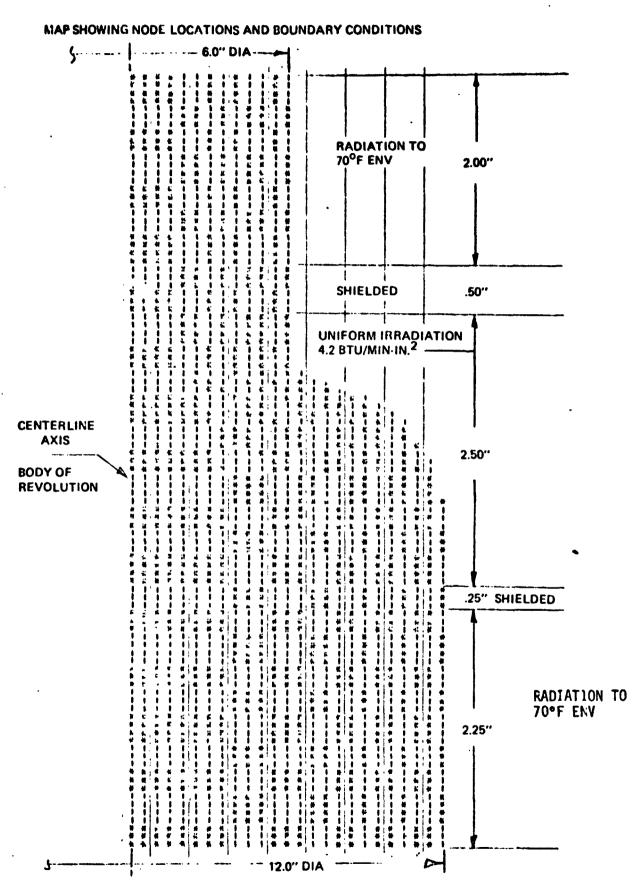


FIGURE: 27 Run 80 BODY OF REVOLUTION NODE MAP

INITIAL CONDITIONS - TIME = 0 HEATING RATE = 551 BTU/LIN COOLING RATE = 772 BTU/LIN 2473⁰F 3073⁰F 2473⁰F 2323^OF

FIGURE: 28. Run 80 - MELT ZONE - TIME - 0

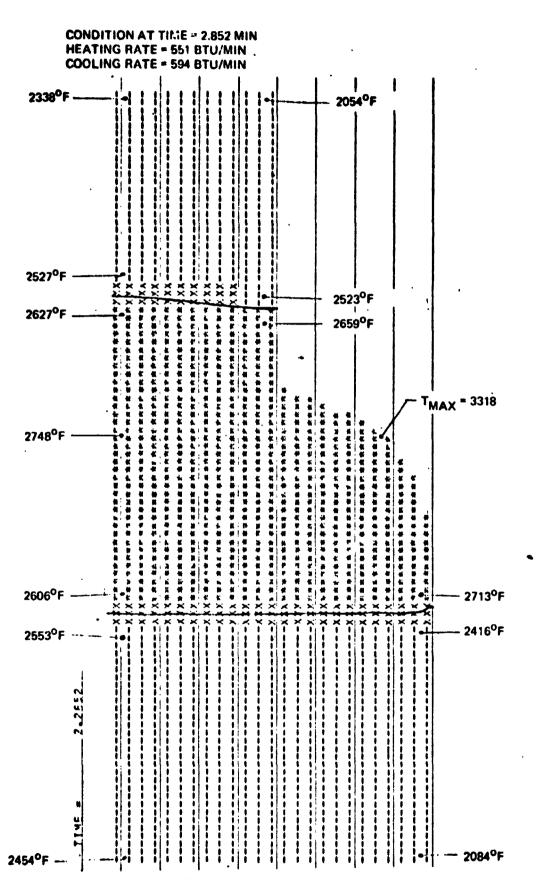


FIGURE: 29. Run 80 - MELT ZONE - TIME = 2.852 MIN.

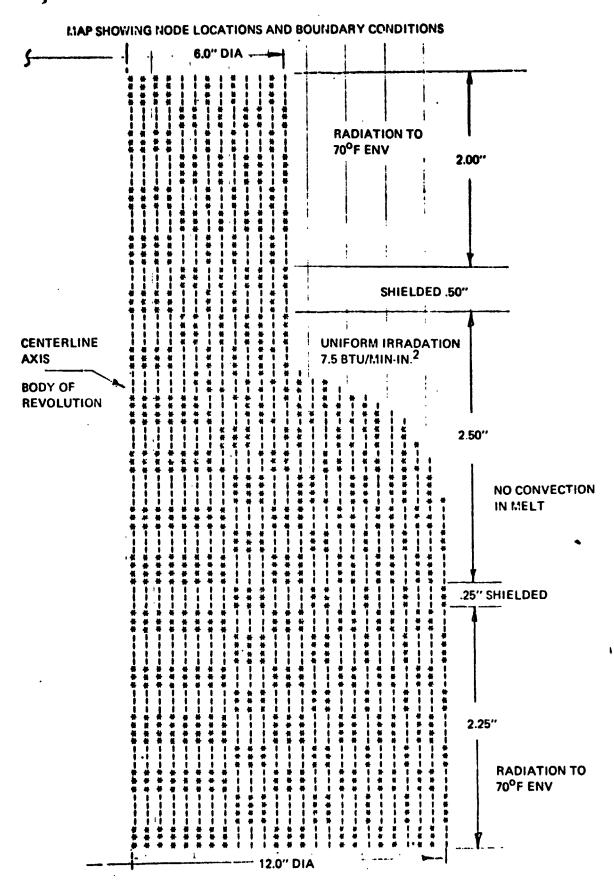


Figure 30. Run 72 - MELT ZONE NODE MAP

HEATING RATE = 984 BTU/MIN COOLING RATE = 1013 BTU/MIN 2563⁰F 2583^OF 2563⁰F

Figure 31. MELT ZONE TIME = 0 RUN 72

INITIAL CONDITIONS - TIME - 0

CONDITION AT TIME = 7.8 MIN

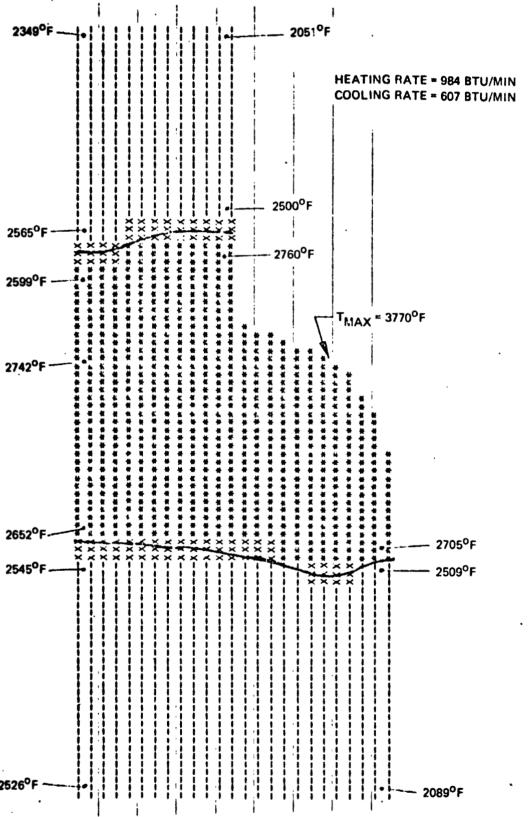


Figure 32. MELT ZONE TIME = 7.8 MIN. RUN 72